S A CR-78

N64 23912 Cot.17



NASA CR-78

NASA CONTRACTOR REPORT

PIGMENT PARTICLE SIZE SEPARATION

by William J. Corbett and Clyde Orr, Jr.

Prepared under Contract No. NAS 8-848 by GEORGIA INSTITUTE OF TECHNOLOGY Atlanta, Georgia for

PIGMENT PARTICLE SIZE SEPARATION

By William J. Corbett and Clyde Orr, Jr.

This report was reproduced photographically from copy supplied by the contractor. Its publication should not be construed as an endorsement or evaluation by NASA of any commercial product.

Prepared under Contract No. NAS 8-848 by GEORGIA INSTITUTE OF TECHNOLOGY Atlanta, Georgia

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For sale by the Office of Technical Services, Department of Commerce, Washington, D.C. 20230 -- Price \$1.25

TABLE OF CONTENTS

		P	age
I.	SUM	MARY	1
II.	INI	PRODUCTION	3
III.	EXF	PERIMENTAL WORK	5
	A.	Description and Operation of Centrifuge	5
	B.	Selection of Titanium Dioxide	<u>1</u> 4
	C.	Dispersion of Titanium Dioxide	14
	D.	Separation of Titanium Dioxide	18
	E.	Washing of Titanium Dioxide	29
IV.	DIS	SCUSSION OF RESULTS	30
٧.	REC	COMMENDATIONS	33
VI.	APP	PENDIX	35
	A.	Centrifuge Construction	36
	В。	Centrifuge Cell Construction	36

LIST OF FIGURES

		Page
1.	Centrifuge with the Cover Removed	6
2.	Centrifuge Cell, Filling Device and Vaned Cap	7
3.	Sedimentation Time versus G's for Various Particle Diameters of Titania	12
4.	Centrifuge Speed versus Number of G's Developed	13
5.	Electron Micrograph of duPont's Experimental Titania, Unseparated	15
6.	Particle Size Distribution of duPont's Experimental Titania, Unseparated	16
7.	Electron Micrographs of Various Particle Size Fractions Obtained	20
8.	Particle Size Distribution of Fraction Number 1	21
9.	Particle Size Distribution of Fraction Number 2	22
10.	Particle Size Distribution of Fraction Number 3	23
11.	Particle Size Distribution of Fraction Number 4	24
12.	Particle Size Distribution of Fraction Number 5	25
13.	Particle Size Distribution of Fraction Number 6	26
14.	Particle Size Distribution of Fraction Number 1 Before and After Refractionation	28
15.	Centrifuge Cell Container	37
16.	Centrifuge Rod End Bearing and Arm	38
17.	Centrifuge Center Block	
18.	Centrifuge Shaft and Cap	
19.	Centrifuge Stand	
20.	Centrifuge Assembly	
21.	Body of Centrifuge Cell	43
22.	Centrifuge Cell Disks	44
23.	Centrifuge Cell Cap	

I. SUMMARY

The objective of this research was to classify titania pigment into a number of size fractions in the diameter of from 0.20 to 2.5 microns and to produce these fractions in experimental quantities of approximately 100 grams.

A batch-type, liquid-phase centrifuge was developed specifically to accomplish the particle size classifications. Its main feature was a cell having a tapered body leading to a special sediment collector. This collector was constructed in several demountable sections so that sedimented titania could be divided into portions simply by isolating the sections. Since the titania was made to settle according to particle size by the centrifuge, the dividing of the sediment into portions resulted in a particle size fractionation of the settled material. A technique was developed for the operation of the centrifuge to ensure satisfactory separation. A technique also had to be developed for dispersing the titanium dioxide as a liquid slurry.

More than 50 grams of six particle size fractions have been produced having 95 per cent of their particles under 2.8, 2.5, 2.4, 1.6, 1.1, and 0.7 microns in diameter, respectively, from a wide-size range (0.1 micron to above 4.0 microns particle diameter) titanium dioxide. Even though completely satisfactory fractionation of the titania cannot be claimed, the classification that was accomplished is good when consideration is given to the wide initial distribution of particle sizes and its less than complete dispersion. In fact, the classification technique developed in this research is probably superior to any other for particles of less than 5 microns diameter.

II. INTRODUCTION

The objective of this research was to classify titanium dioxide pigment into a number of size fractions in the diameter range of from 0.20 to 2.5 microns and to produce these fractions in approximately 100 gram quantities. It was desired also that the titania be of the rutile structure and that its particles be relatively spherical in shape.

Particle size classification is usually accomplished with the particles suspended in either a gas or a liquid. Gas-phase operation would be preferable for small-particle separations because this permits utilization of the greatest density difference between the particles and the suspending medium if particles did not tend to stick together, or agglomerate. As it turns out, gas-phase classification may be employed only with those powders that are relatively easily dispersed, i.e., each and every particle freed from attachment with all others. If the particles are not readily dispersed, liquid phase operation is required, since, by doing so, both chemical dispersing agents and high shearing forces may be utilized to separate one particle from another. Neither an agent nor high shear can be readily employed with gas-phase operation.

Titanium dioxide pigment falls in the class of powders that are dispersed with difficulty. For this reason, separation in the liquid phase was attempted first, was studied most intently, and was adopted for the final separation. Gas-phase operation was tested, but was never investigated extensively because nothing like complete dispersion

could be obtained. Numerous liquid-phase centrifuges and modifications of them were investigated in the first year of research effort. These instruments were of both commercial and original design. The most satisfactory separations finally were obtained with a batch-type, liquid-phase centrifuge constructed especially for this effort.

^{1.} Clyde Orr, Jr., Andrew McAlister, and W. J. Corbett, Annual Report No. 1, Georgia Tech Research Project No. A-542, NASA Contract No. NAS8-848.

III. EXPERIMENTAL WORK

A. Description and Operation of Centrifuge

On the basis of the first year's research a batch-type, liquidphase centrifuge constructed especially for this effort was selected as the most promising technique for obtaining the desired particle-size separations. Figure 1 is a photograph of the centrifuge with the 1/2inch-thick plywood top removed. The unit consists basically of two cylindrical containers mounted on a balanced rotor approximately 30 inches from the center of rotation. The cylindrical containers are holders for settling chambers in which a slurry of the material to be size-fractionated is contained. Detailed engineering drawings of the centrifuge are presented in the Appendix. A 5-horsepower electric motor coupled through a variable speed controller powers the rotor. A 3-phase voltage supply system utilizing two variable transformers permits the centrifuge to be started and brought to operating speed very slowly. The centrifuge was designed for operational speeds of up to 750 revolutions per minute and has a design limit of 1100 revolutions per minute. Two centrifuge cells similar to the one shown in Figure 2 are employed as the actual slurry holders for a size separation. Detailed engineering drawings of the centrifuge cell are presented in the Appendix also. This cell is an adaptation of a design by Whitby of a very small centrifuge cell to be used in particle size-distribution analyses. The present cell, constructed of Plexiglas, is 13-1/2 inches long with an

^{2.} K. T. Whitby, "A Rapid General Purpose Centrifuge Sedimentation Method for Measurement of Size Distribution of Small Particles," Heating, Piping and Air Conditioning 61, 33-47 (1955).

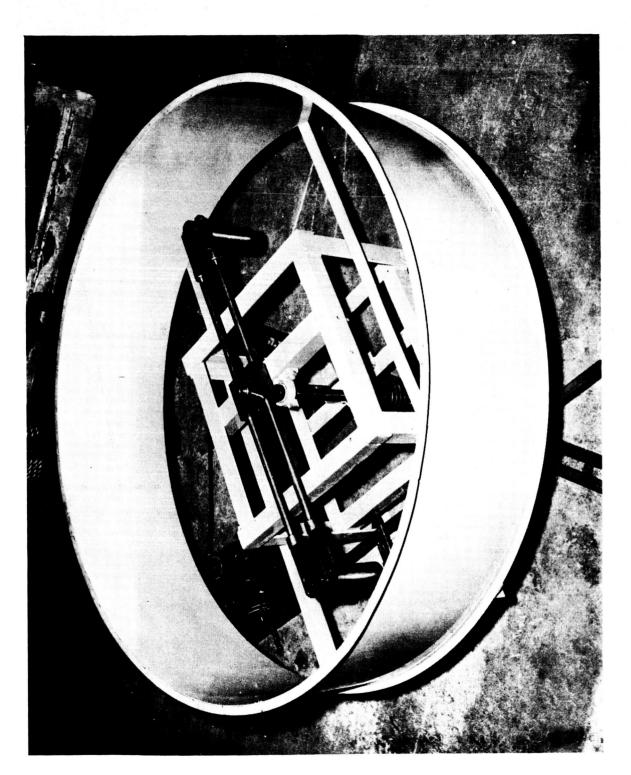


Figure 1. Centrifuge with the Cover Removed.

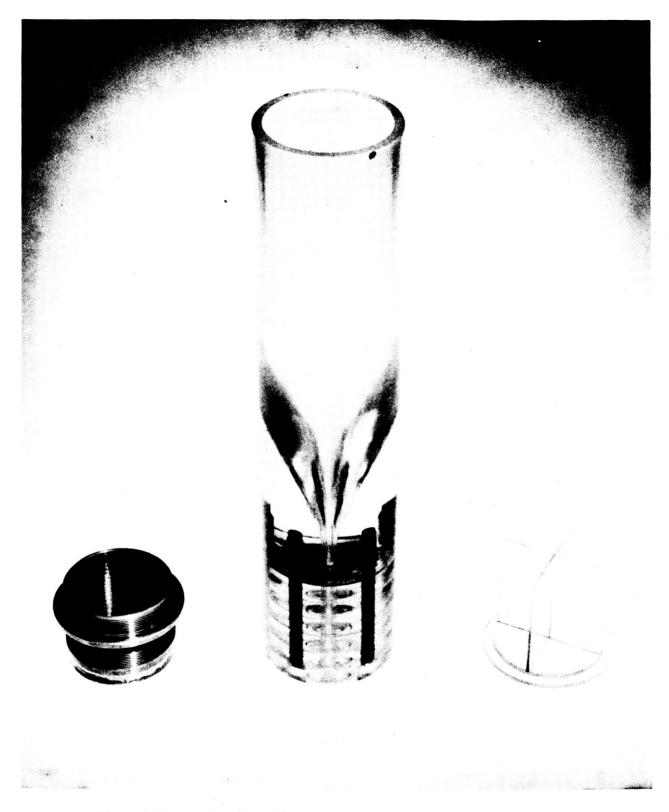


Figure 2. Centrifuge Cell, Filling Device and Vaned Cap.

inside diameter that tapers from 2-1/2 inches to 3/16 inch. The 3/16-inch portion extends through five discs that are secured to the body of the cell with four Allen-head machine screws. The cylindrical containers that hold these cells swing outward during operation so that particles in the cells are forced horizontally outward. The motion of the particles is governed by Stokes law and, after a given amount of time, this motion establishes a gradient of particle sizes along the axis of the centrifuge cell with the larger particles being at the outer radius, i.e., nearer the cell bottom. If the centrifuge is allowed to operate for a sufficient length of time these particles will be forced into the narrow portion of the centrifuge cell and compacted into a semirigid mass. The particles within this mass will also be distributed in a size-gradient with the largest particles at the bottom and the smallest at the top.

The entire operation, being merely an accelerated gravity technique, is straightforward; however definite experimental procedures had to be followed to obtain the most complete particle size separation. For cleanest separation, it is necessary that all particles start at or near the same level in the suspending fluid. Furthermore, to attain capacity as much material as possible must be handled, but the slurry must not be so concentrated that significant particle agglomeration occurs during sedimentation. The requirement of all particles starting at or near the same point in the centrifuge cell is met by introducing the particles to be separated as a slurry in a thin layer at the top of the cell. The cell is filled initially with approximately 500 ml of water with 0.5 per cent of a suitable surfactant added. Approximately

30 to 60 ml of a slurry of the material to be size-fractionated are placed on the water as a distinct layer with the aid of the filling device shown to the left of the centrifuge cell in Figure 2. This device consists of a hollow metal cylinder the outer diameter of which is slightly less than the inner diameter of the centrifuge cell; it has a fine-mesh screen across the bottom. A metal collar adjustable by screw threads is fitted to the outside of the cylinder. When inserted in the centrifuge cell the position of the collar is set so that the screen just breaks the surface of the water in the cell. The slurry that has previously been prepared with a liquid having a lower density than water, say methanol and water, is then carefully poured into the filling device and the device is slowly withdrawn. This technique allows the slurry to be placed as a separate layer on the surface of the water with a minimum of mixing between the two. The exact amount of slurry added depends on its initial concentration, sufficient slurry being required exactly to fill the lower portion of the centrifuge cell With separated material when the sedimentation has been completed. The concentration of particle material in the slurry is usually 5 to 10 per cent by weight. The overall concentration of particle material in the cell during sedimentation is usually only 0.6 to 0.7 weight per cent.

After the filling device has been removed a vaned cap is placed in the mouth of the cell. One such cap is shown to the right of the centrifuge cell in Figure 2. This cap serves a twofold purpose. The vanes retard rotation of the slurry layer during start-up of the centrifuge which would mix the two layers, and the cap prevents evaporation of

liquid during the operation of the centrifuge. The latter is especially important because evaporation could cause a thermal gradient along the axis of the cell and would disturb the sedimentation.

The centrifuge is started as soon as possible after the cells are The power is switched on with the variable speed controller set at its slowest operating position (a centrifuge rotor speed of approximately 150 revolutions per minute) and the variable voltage transformers The centrifuge rotor is then started by a gradual increase of voltage to the driving motor through the variable voltage transformers. The voltage is increased at such a rate as to bring the centrifuge cell containers to an essentially horizontal position in approximately 10 to 12 revolutions of the centrifuge rotor. Experience has indicated that this starting rate produces the least disturbance of the slurry layer. A faster starting rate causes the more dense water layer to move under and displace the slurry layer with subsequent mixing. Since the vaned caps do not completely eliminate the swirling motion imparted to the liquid by the centrifuge rotation, a slower starting rate allows mixing to occur between the two layers due to this imparted motion.

After the centrifuge has been brought to the speed that causes the containers to be nearly horizontal, the lid of the centrifuge is closed and the variable transformers are advanced until the full line voltage is supplied to the motor. At this point the mechanical speed controller (a Speed-Ranger, Master Power Corp., Solon, Ohio) is also advanced until the desired operating speed is attained. This speed is predicted by the number of G's (multiplies of the acceleration due to gravity) required

to achieve complete sedimentation of the particles being separated in a reasonable length of time. The required number of G's can be estimated from the Stokes law relationship for the settling of particles if particle density and range of sizes are known for the material being fractionated. This relationship may be expressed in the form

$$G = \frac{18 L \mu}{980 p^{2} (\rho_{p} - \rho) t}$$
 (1)

where G is the number of multiples of acceleration due to gravity, L is the length of sedimentation distance in the centrifuge cell, μ is the viscosity of the fluid in which sedimentation is occurring, ρ is the fluid density, ρ_p is the density of the particle, t is the time of settling, and D_p is the particle diameter. Figure 3 is a plot of this relationship for various sizes of titanium dioxide particles settling in water. Figure 4 is a plot of centrifuge rotor speed versus the number of G's developed at the mid-point of the length of the centrifuge cell.

When the centrifugation has been completed the cells are removed from the containers and the liquid poured off. Since the sedimented particles are present in the collector section of the cell as a semirigid mass they remain in the cell. The lower portion of the cell is then disassembled, each disk containing a slug of material with particles in a specific portion of the initial particle size distribution. These slugs are then forced from the collector disks by means of a polished metal rod just slightly smaller than the diameter of the hole in the collector, and these slugs are collected in a suitable container. Since the cell has five removable disks all 1/2-inch thick, six fractions of

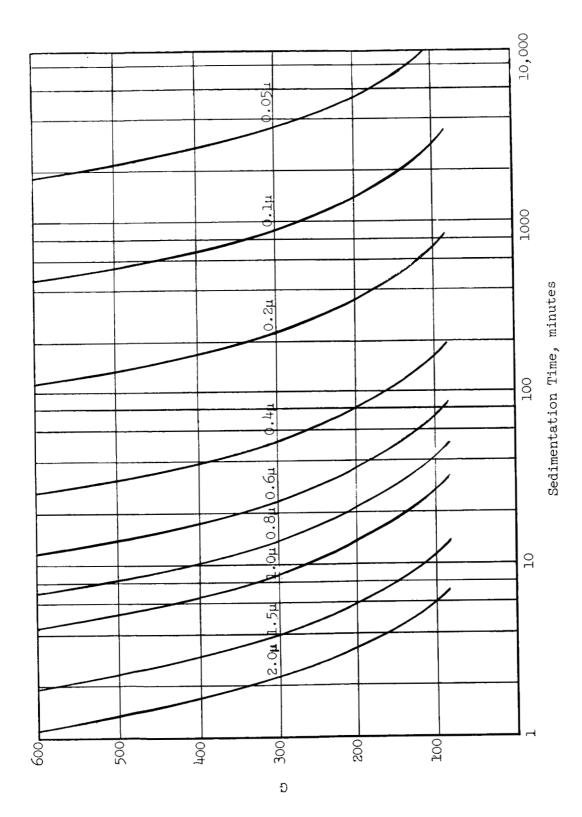


Figure 3. Sedimentation Time versus G's for Various Particle Diameters of Titania.

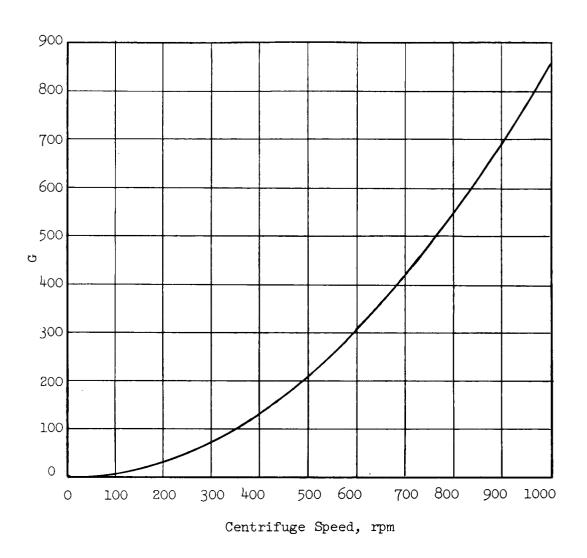


Figure 4. Centrifuge Speed versus Number of G's Developed.

material can be obtained if sufficient material is introduced originally to bring the final deposit height 1/2 inch into the main cell body.

B. Selection of Titanium Dioxide

The principle objective of this project was to classify titania into a number of particle size fractions in the range 0.20 to 2.5 microns diameter. It was also desired that this pigment have the rutile structure and be composed of spherical or nearly spherical particles. Finding a titanium dioxide with which these specifications could be met was difficult and not entirely achieved. Commercially available titania pigment does not have a satisfactorily wide particle diameter range. These pigments normally range from 0.1 to 0.7 micron in diameter. Inquiries were even made of British producers since it was understood a number of them were still using the sulfate process for manufacturing titania pigment, and this process usually produces a larger particle size than the gaseous process employed by American producers. However, they too were unable to supply a satisfactory pigment. finally chosen for particle-size classification, was obtained in late March 1962, through the sponsor from E. I. duPont deNemours & Company. This titania, an experimental material designated 4087-92-WS, had particles from less than 0.1 micron to greater than 4.0 microns in diameter. An electron micrograph of some of this material is presented in Figure 5 and the particle size distribution is presented in Figure 6.

C. Dispersion of Titanium Dioxide

The success of the separation technique developed with the batch-type

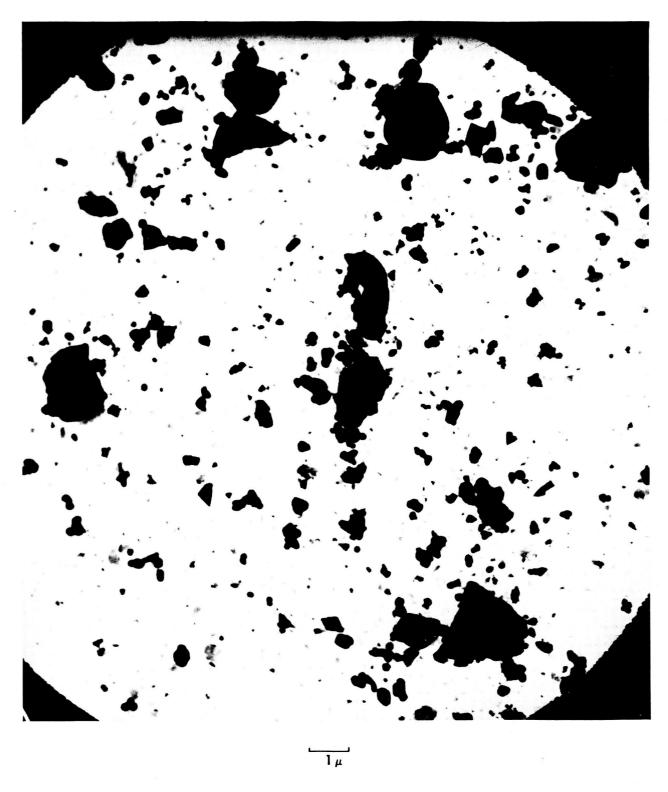


Figure 5. Electron Micrograph of duPont's Experimental Titania, Unseparated.

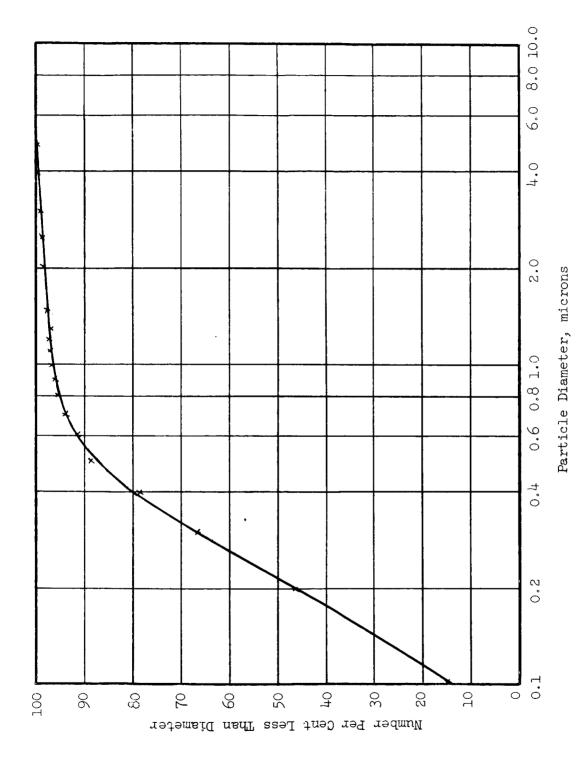


Figure 6. Particle Size Distribution of duPont's Experimental Titania, Unseparated.

liquid-phase centrifuge depends on complete dispersion of the pigment as individual particles. This is most readily accomplished by dispersing the particles as a liquid slurry, and introducing them into the centrifuge cell is more convenient if they are suspended as a slurry. However, considerable research was necessary to arrive at a technique for producing a satisfactory dispersion. The adopted technique utilized a 1.2 quart porcelain ball mill. The mill was charged with 75 grams of titania, 75 ml of a 2:1 mixture of methanol and water, 7.5 ml of a surfactant (Tamol 731-25%) and 1 kg of flint pebbles. This charge was ground for 8 to 12 hours at 115 revolutions per minute. After grinding, the flint pebbles were removed and an additional 675 ml of 2:1 methanol and water mixture was added. This produced a slurry with a specific gravity of 0.94 to 0.96 and a titania concentration of approximately 8 weight per cent. Before fractionation of the freshly prepared slurry the specific gravity was measured carefully and two 10 ml aliquots of the slurry were evaporated to dryness to obtain the exact concentration of titania. Knowledge of the exact weight concentration of titania was necessary to determine precisely how much slurry to add to each centrifuge cell to insure the correct depth of deposit after fractionation. This quantity can be obtained from knowledge of the slurry concentration, the volume of the collector section of the centrifugation cell, the density of titania, and the packing fraction of the compacted particles. This latter quantity was determined experimentally to be approximately 0.53, i.e., 53 per cent of the available volume in the collector was occupied by titania particles.

On the basis of experiment and the best available information, Tamol 731-25%, a surfactant manufactured by Rohm and Haas, Philadelphia, Pennsylvania, appears to be the most satisfactory dispersant for titania in a methanol-water system. Flint pebbles were employed as the grinding media rather than porcelain cylinders because of their greater hardness and resistance to attrition. The use of porcelain cylinders appeared to introduce some very small flakes of the porcelain into the titania.

The titania slurries used in this work were prepared in the quantity mentioned above only for convenience. There does not appear to be any reason why the slurry could not be prepared in larger quantities provided the relative ratios of the variables remained the same.

D. Separation of Titanium Dioxide

The duPont experimental titania was fractionated with the batch-type, liquid-phase centrifuge operating at 500 revolutions per minute (200 G's) for 5-1/2 to 6 hours. After collection of approximately 50 grams of each of the six fractions resulting from the classification, the fractions were dried for several hours at 125°C, were ground lightly with a mortar and pestle to break up any large lumps of material, and samples were taken for examination with the electron microscope. The samples were prepared for electron microscope examination by placing a small quantity of the titania on a glass microscope slide with a few drops of an amyl acetate and collodion solution, placing another glass microscope slide on top, and mulling the sample by moving the two slides. When the sample had been sufficiently mulled the top slide was removed by sliding it off the lower one. This left the sample in a film on the bottom slide. This film, which dried quite rapidly, was sectioned with a razor blade and a small section floated onto

a still water surface. The electron microscope grid, held in a pair of tweezers was next brought up under this film and the film and grid lifted from the water. The grid was then ready for insertion into the electron microscope.

Electron micrographs were obtained for each of the fractions obtained and are presented in Figure 7. The fraction numbered 1 is from the lowest disk in the collector section and contains the largest particles. The fractions are numbered progressively up to number 6, which is the last fraction sedimented and contains the smallest particles collected. A definite gradation in particle size is apparent from only a qualitative visual examination of these micrographs. However, even though the fractions containing the smaller particle sizes are quite free from the larger particles, the fractions containing the larger particles do contain some of the smaller particles. This fact is even more apparent from the particle size distributions that were obtained from these and other micrographs as presented in Figures 8 through 13. It should be emphasized that these distributions are on a count or-number-of-particles basis rather than a weight basis. On a weight basis the percentage contribution of the very small particles would be quite small in a distribution such as that for fraction number 1 rather than the large percentage of the distribution that they represent on a number basis.

Between 50 and 60 grams of each of the fractions represented in Figures 8 through 13 were collected. After this initial separation, fraction number 1, which contained an unusually larger percentage of small particles even though the percentage of large particles had been increased significantly, was refractionated using a technique similar to that used

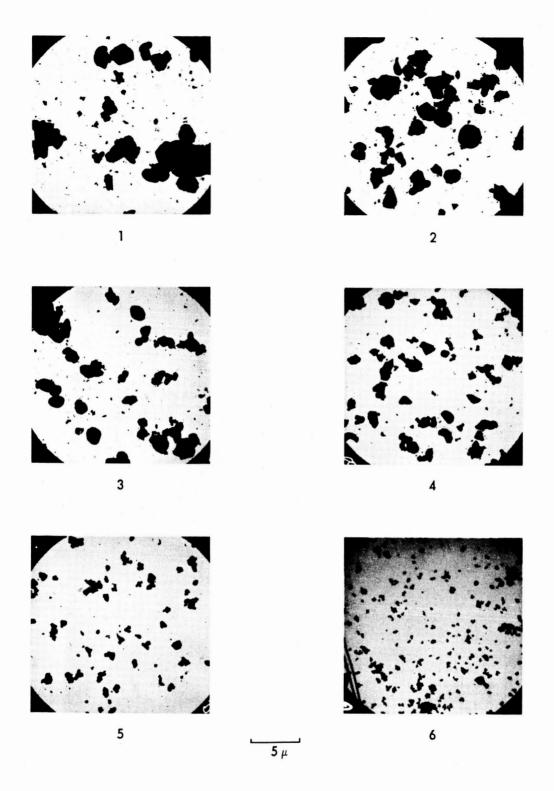


Figure 7. Electron Micrographs of Various Particle Size Fractions Obtained.

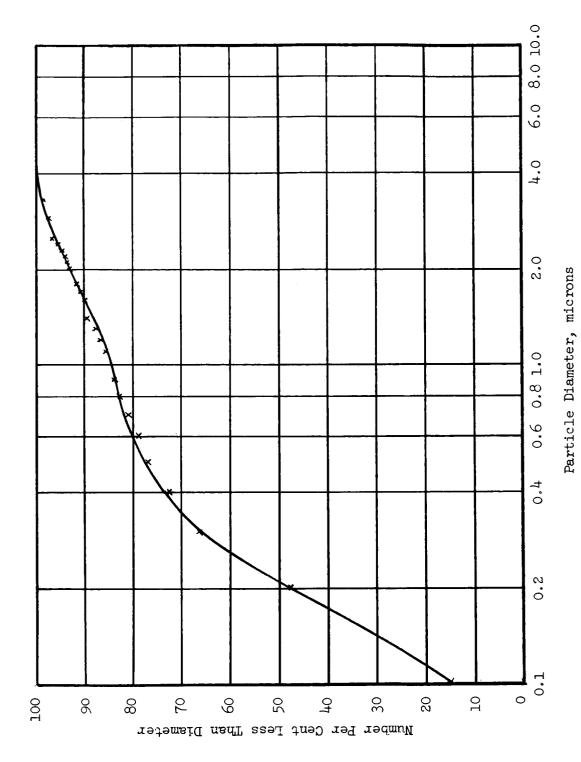


Figure 8. Particle Size Distribution of Fraction Number 1.

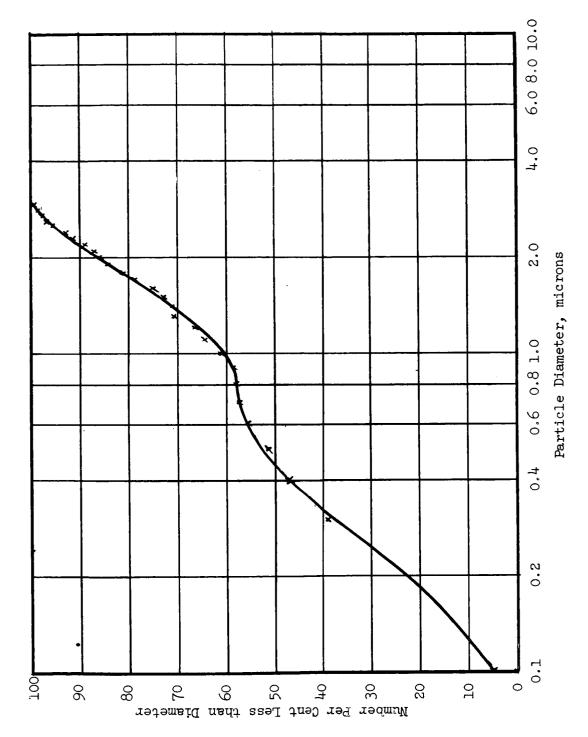


Figure 9. Particle Size Distribution of Fraction Number 2.

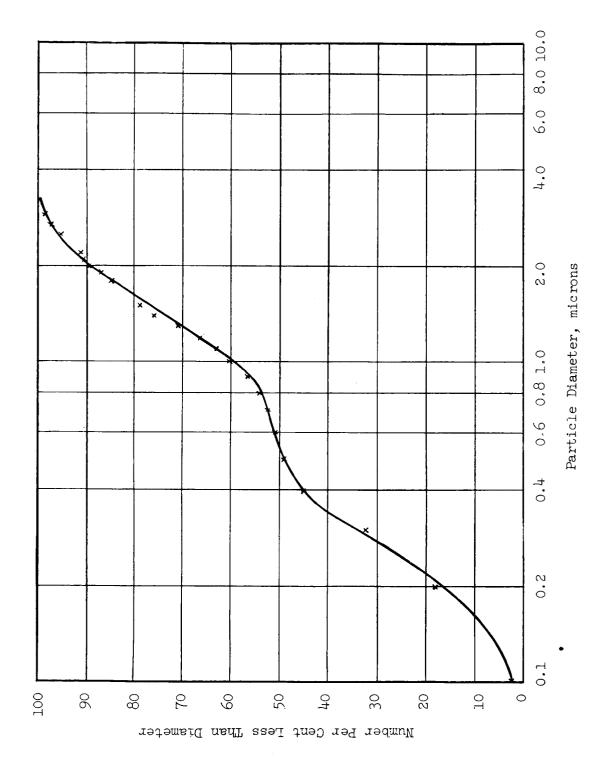


Figure 10. Particle Size Distribution of Fraction Number 3.

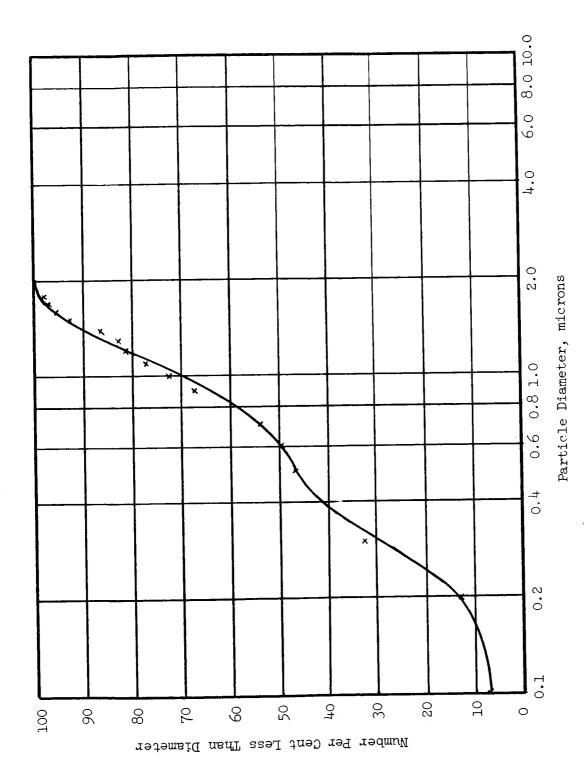


Figure 11. Particle Size Distribution of Fraction Number 4.

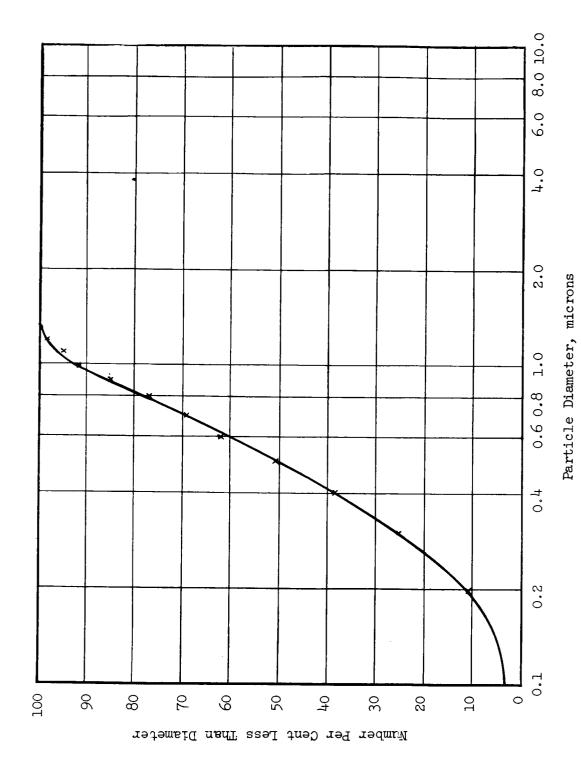


Figure 12. Particle Size Distribution of Fraction Number 5.

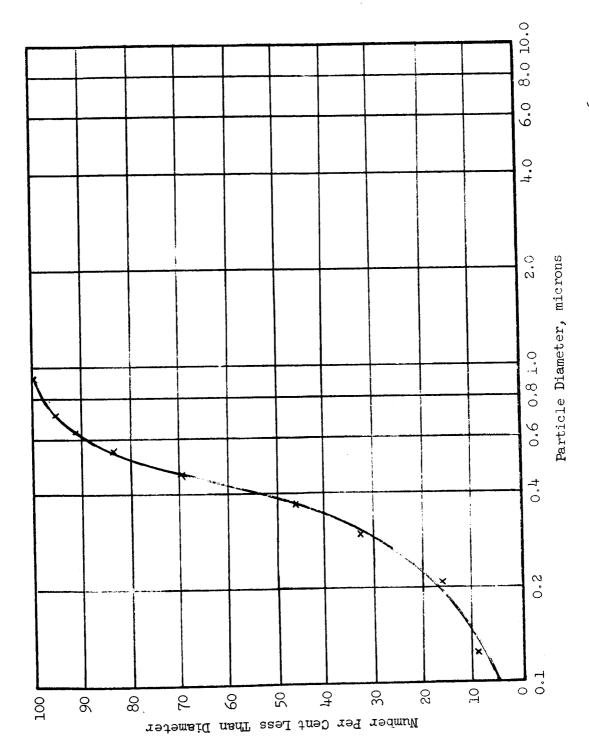


Figure 13. Particle Size Distribution of Fraction Number 6.

for the initial separation. This material was dispersed in the same manner as the original material. However, instead of requiring a 6-hour centrifugation to obtain six fractions as before, sufficient slurry was added to the cell to fill the collector section with just the large (above one micron) particles and the centrifuge operated for approximately 30 minutes at 200 revolutions per minute. Therefore, all the material in the collector section of the centrifuge cell was treated as one fraction because the small particles that had not been sedimented were poured off in the supernatant liquid. This second separation, of course, was done in an attempt to reduce the percentage of small particles in this sample. The results of this separation are shown in Figure 14. As can be seen, the percentage of large particles was again increased significantly; however, the distribution is still bimodal with a large number of small particles present.

A "standard," finally, was prepared from the original material to be available for comparison purposes. Approximately 200 grams of the duPont experimental titania was dispersed in the exact manner as for the size classifications, including dilution to an approximately 8 weight per cent slurry. The methanol and water were removed from this titania with a Delaval centrifugal laboratory clarifier of the Delaval Separator Company, Poughkeepsie, New York. Approximately 165 grams of material were recovered. This was dried and the large lumps crushed in a mortar and pestle as was done for the separated fractions. This treatment did not alter the size distribution of the material but merely insured that it was treated in the same manner with respect to addition to surfactant, etc., that the classified fractions were.

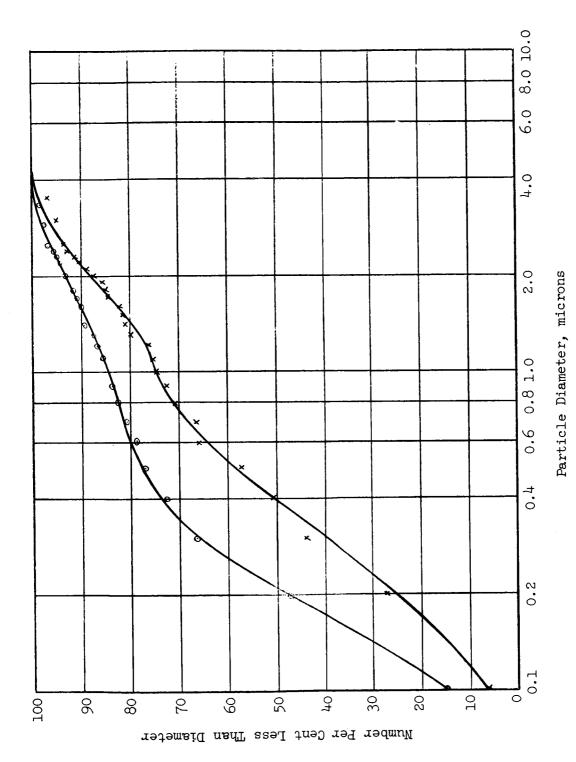


Figure 14. Particle Size Distribution of Fraction Number 1 Before and After Refractionation.

E. Washing of Titanium Dioxide

Time did not permit washing the collected pigment free of the surfactant used in the slurry preparation although several means of accomplishing this were critically examined. It appears that this could best be accomplished by washing the titania in an adequate volume of water and centrifuging the material with the batch-type, liquid-phase centrifuge to collect the titania. Here the centrifuge is merely being used to concentrate the titania rather than to classify it and the need for using only a layer of slurry at the start and having just the exact amount to fill the collector section are eliminated. The surfactant washed from the titania is, of course, poured off with the water at the end of the centrifugation. Both filtration and continuous centrifugation, such as is possible with the DeLaval clarifier, were investigated as means for recovering the titania from the water required to wash out the surfactant. Both membrane and fiber filters were examined. However, when these filters had built up a filter cake sufficient to prevent the passage of titania particles, the pressure drop across the filter was so great that the filtration rate was reduced to practically nothing. The DeLaval clarifer would satisfactorily concentrate the titania from the wash water for the samples containing the coarser particles, but the efficiency for the very fine fractions is questionable. In any event, the loss of material involved in utilizing the DeLaval clarifier would be excessive for the small samples available.

IV. DISCUSSION OF RESULTS

The batch-type, liquid-phase centrifuge developed for this project performed very satisfactorily. The particle size classifications obtained with it, while not as sharp as desirable, were superior to any that were obtained with any other devices tested in the earlier phases of the program.

The principal difficulty encountered during this research was not the development of a satisfactory separating device, but was obtaining a titania with satisfactory characteristics. The experimental titania that was finally used was not entirely satisfactory due to the distribution of particle sizes and the fact that the particles were only approximately spherical. The poor distribution of particle sizes (i.e., 97 per cent-by-count below 1 micron) probably contributed more to the lack of success in obtaining more narrow size classifications than any other single factor. This disproportionate amount of finer particles made the normally difficult problem of separating the fines from the coarse material just that much more difficult.

The exact reason for the occurrence of fine particles in the fractions that were supposed to contain only the large particles is not clear. Either the dispersion of particles was incomplete, leaving some of the fines adhering to the large particles or the large particles intercepted the fines in passing through the slurry layer and dragged them along. Since the only real way to evaluate the degree of dispersion attained in a slurry is to attempt a separation and determine whether or not the particles are classified, this question was not resolved. In this case

the extremely large number of fines made the possibility of interception by the larger particles a very important factor. However, a close examination of the particle size distributions presented in Figures 8 through 13 suggests that the presence of the fines in the samples containing the larger particles is due primarily to adherence of the fines to the coarse particles. This is indicated by the nature of the bimodal distribution of samples 1 through 4. As the size of the largest particles present in the samples decreases, the percentage of the fine particles comprising the lower portion of the bimodal distribution decreases also. This would be expected since the decreasing particle size of the coarse fraction offers less and less total surface for adherence of the very fine particles and consequently fewer fines are present. When the largest particles are less than 1.5 microns in diameter, as in samples 5 and 6, and there are fewer large particles for the very fine ones to adhere to, the bimodal nature of the curve disappears and the separation becomes much sharper.

The overlap of particle sizes between fractions 5 and 6 is probably due to the fact that the quantity of particles with sizes between 1.5 microns and approximately 0.5 or 0.6 micron was not sufficient to fill the collector disk completely and some of the material with smaller sizes began filling the disk until the sediment height was sufficient for the particles to deposit in the section in which fraction number 6 was collected. This same condition, where the weight fraction of a particular particle size range was greater than could be accommodated in a single disk, also explains why fractions 3 and 4 had almost identical

particle size distributions. Actually, the ideal situation for a separation where fractions containing only certain particle sizes are desired would be to calculate the disk thicknesses required for the collector section rather than to use disks all the same thickness. This technique would require a determination of the particle size distribution, on a weight basis, of the original material, calculation of the volumes that the desired fractions of a certain size sample would occupy when sedimented, and construction of the various disks of the collector section to suit these volumes. This, of course, is a more refined version of the technique developed in this work and there was not adequate time to utilize it here.

The attempt to sharpem the distribution of fraction number 1 by refractionation was moderately successful. The improvement in the distribution was significant enough to indicate that four or five successive fractionations might reduce the unwanted fine particles to a negligible amount. However, the necessary handling of the material in a refractionation causes a loss of approximately 15 per cent.

V. RECOMMENDATIONS

Since the equipment and basic techniques for accomplishing particle size fractionation in the very small particle size ranges have been extended in this research, the following recommendations are offered for further investigation of improvements into small size classifications. It appears that the area where improvement would yield the greatest benefit is in dispersion of the titanium dioxide. Before any significant improvement can be made in the classification, ther number of fines adhering to the larger particles must be reduced. Since it is felt that liquid-phase ball milling offers as good a system for producing the necessary shear as any, probably the most significant improvement in the dispersion technique would come from utilizing a liquid that would transmit the shear more effectively than methanol and water. A more viscous liquid, for example, would permit this. Glycerol, which is miscible with water, might prove satisfactory. However, some other diluent besides water would be required because the specific gravity of the slurry must be less than that of the sedimenting medium. Alcohol could be used, but the glycerol-alcohol system might require a different surfactant from the one used with methanol and water.

Any future attempts to obtain specific size fractions from a particular distribution of particle sizes should be conducted with a centrifuge cell using disks of calculated thicknesses rather than an arbitrarily chosen thickness as was done in this research. The technique for calculating the proper disk thickness was outlined in the preceding section.

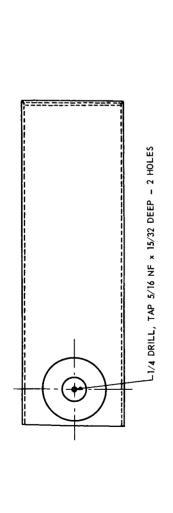
VI. APPENDIX

A. Centrifuge Construction

Figures 15 through 20 present the necessary engineering drawings for construction of a batch-type, liquid-phase centrifuge such as used in this research. In addition to the information provided in these drawings the following details are provided: (1) the shield for the centrifuge was fabricated from 10 gauge mild steel and was 75 inches in diameter, (2) the shield was strengthened with two hoops rouled from 1-inch mild steel angle placed at the top and bottom edges, (3) the top for the centrifuge was constructed from 3/4-inch plywood and fitted with a cabinet latch on the front edge of the hinged portion, (4) the pillow blocks for the centrifuge shaft were Fafnir LAK with a 1-7/16-inch bore, (5) the drive pulley on the shaft was a 3-groove, "B" pulley with a 5.6-inch pitch diameter.

B. Centrifuge Cell Construction

Figures 21 through 23 contain the necessary engineering drawings for construction of the centrifuge cells for use with the batch-type, liquid-phase centrifuge. The tapered portion of the centrifuge cell body shown in Figure 20 is an arbitrary curve and a tool to cut this portion must be fabricated. The curve shown on the drawing should be scaled to provide a template for the tool; however, the curve is not too critical. The O-rings used in the assembly were 1/2-inch inside diameter, 5/8-inch outside diameter and 1/16-inch thick.



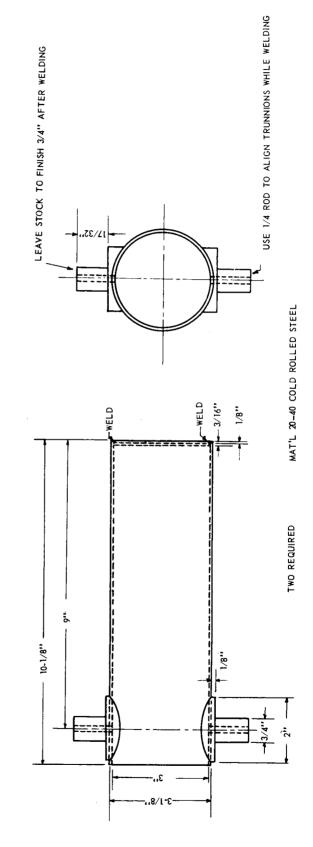


Figure 15. Centrifuge Cell Container.

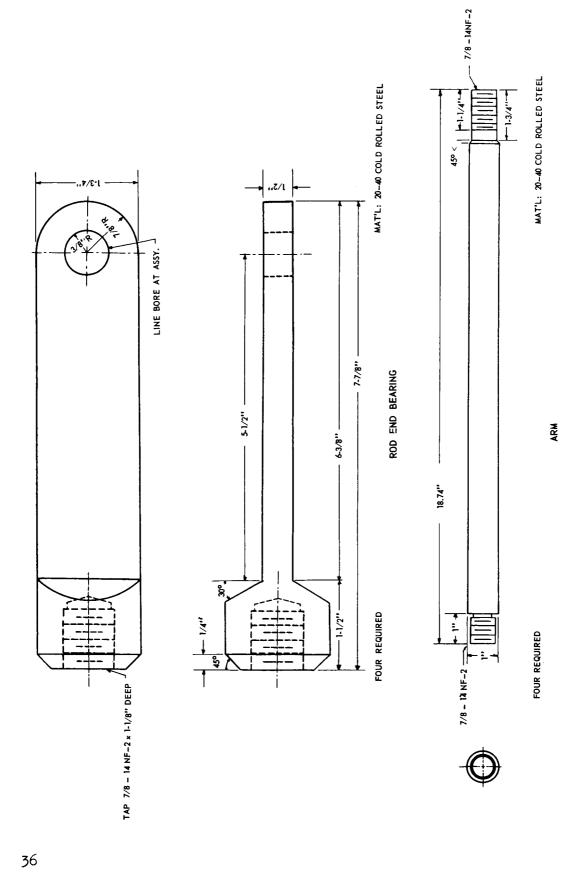
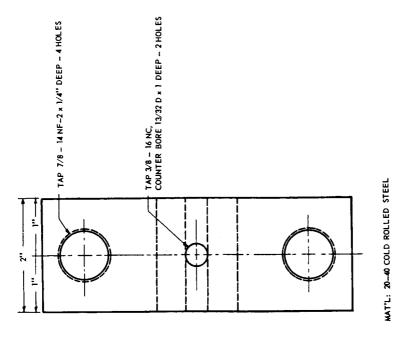
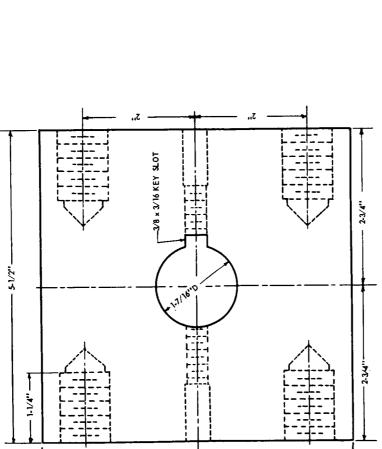


Figure 16. Centrifuge Rod End Bearing and Arm.





..2/1-5

Figure 17. Centrifuge Center Block.

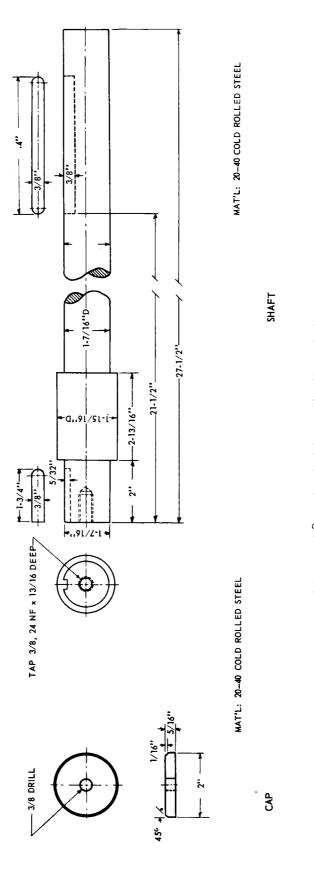


Figure 18. Centrifuge Shaft and Cap.

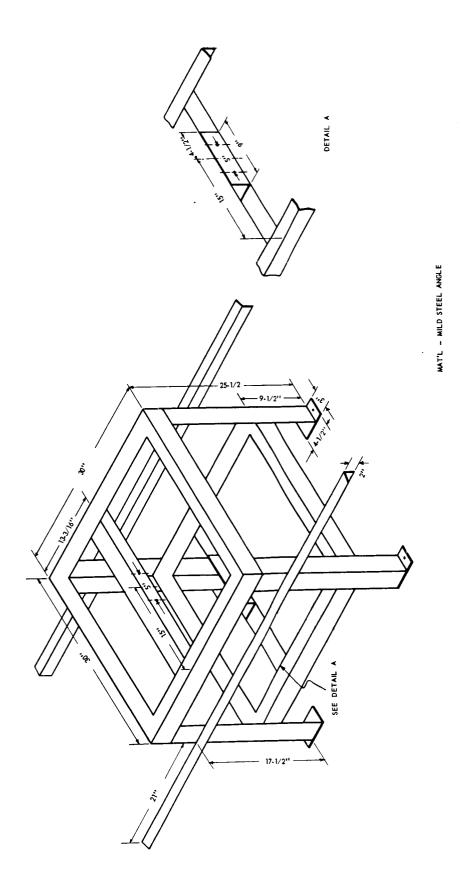


Figure 19. Centrifuge Stand.

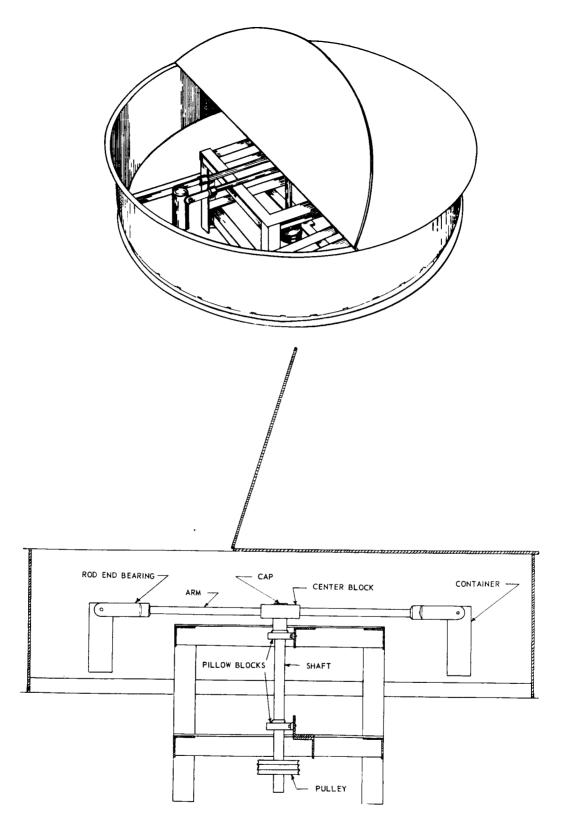
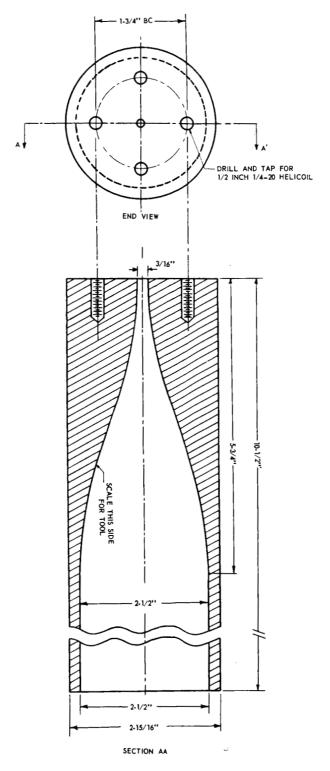


Figure 20. Centrifuge Assembly.



MAT'L: PLEXIGLASS

Figure 21. Body of Centrifuge Cell.

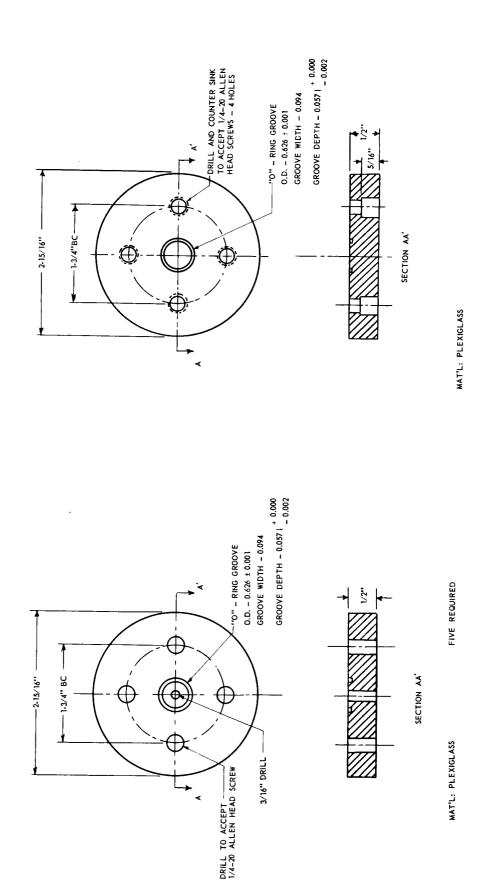
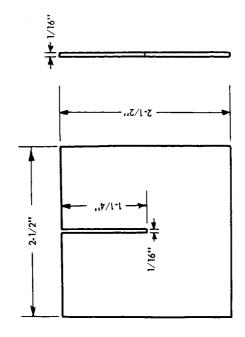


Figure 22. Centrifuge Cell Disks.

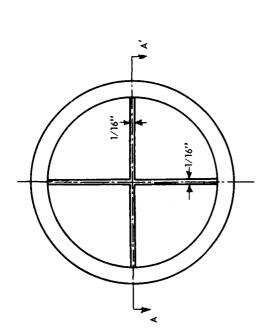




TWO REQUIRED

MAT'L: PLEXIGLASS

MAT'L: PLEXIGLASS



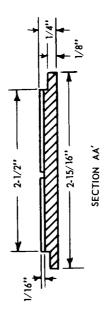


Figure 23. Centrifuge Cell Cap.